Common Market for Eastern and Southern Africa

EDICT OF GOVERNMENT

In order to promote public education and public safety, equal justice for all, a better informed citizenry, the rule of law, world trade and world peace, this legal document is hereby made available on a noncommercial basis, as it is the right of all humans to know and speak the laws that govern them.

COMESA 250 (2006) (English): Wool- Determination of mean diameter of fibres - Air permeability method

ISO INSIDE

BLANK PAGE

BRUTUM FULMEN

IGNORANTIA JURIS NON EXCUSAT

PROTECTED BY COPYRIGHT

COMESA HARMONISED STANDARD

COMESA/FDHS 250: 2006

Wool- Determination of mean diameter of fibres - Air permeability method

REFERENCE: FDHS 250: 2006

Foreword

The Common Market for Eastern and Southern Africa (COMESA) was established in 1994 as a regional economic grouping consisting of 20 member states after signing the co-operation Treaty. In Chapter 15 of the COMESA Treaty, Member States agreed to co-operate on matters of standardisation and Quality assurance with the aim of facilitating the faster movement of goods and services within the region so as to enhance expansion of intra-COMESA trade and industrial expansion.

Co-operation in standardisation is expected to result into having uniformly harmonised standards. Harmonisation of standards within the region is expected to reduce Technical Barriers to Trade that are normally encountered when goods and services are exchanged between COMESA Member States due to differences in technical requirements. Harmonized COMESA Standards are also expected to result into benefits such as greater industrial productivity and competitiveness, increased agricultural production and food security, a more rational exploitation of natural resources among others.

COMESA Harmonized Standards are developed by the COMESA experts on standards representing the National Standards Bodies and other stakeholders within the region and are approved after circulating Final Draft Harmonized Standards (FDHS) to all member states for at least three months. The assumption is that all contentious issues would have been resolved during the previous stages or that an international or regional standard being adopted has been subjected through a development process consistent with accepted international practice.

COMESA Standards are subject to review, to keep pace with technological advances. Users of the COMESA Harmonized Standards are therefore expected to ensure that they always have the latest version of the standards they are implementing.

This COMESA standard is technically identical to ISO 1136:1976.- Wool- Determination of mean diameter of fibres - Air permeability method

A COMESA Harmonized Standard does not purport to include all necessary provisions of a contract.
Users are responsible for its correct application.

INTERNATIONAL STANDARD 1136

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION •МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ •ORGANISATION INTERNATIONALE DE NORMALISATION

Wool — Determination of mean diameter of fibres — Air permeability method

Laine — Détermination du diamètre moyen des fibres — Méthode perméamétrique

First edition — 1976-11-15

UDC 677.31/.34.017.224.3

Ref. No. ISO 1136-1976 (E)

Descriptors : natural fibres, animal fibres, wool fibres, dimensional measurement, diameters.

Price based on 8 pages

ISO 1136-1976 (E)

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 38, *Textiles*, has reviewed ISO Recommendation R 1136-1969 and found it technically suitable for transformation. International Standard ISO 1136 therefore replaces ISO Recommendation R 1136-1969, to which it is technically identical.

ISO Recommendation R 1136 had been approved by the member bodies of the following countries :

Australia
Belgium
Brazil
Canada
Cuba
Czechoslovakia
Denmark
Egypt, Arab Rep. of
France
Germany
Greece
Hungary
India
Iran
Israel
Italy
Japan
Korea, Rep. of
Netherlands
New Zealand
Norway
Poland
Portugal
Romania
South Africa, Rep. of
Spain
Sweden
Switzerland
Turkey
United Kingdom
U.S.S.R.

No member body had expressed disapproval of the Recommendation.

No member body disapproved the transformation of the Recommendation into an International Standard.

© **International Organization for Standardization, 1976 •**

Printed in Switzerland

Wool — Determination of mean diameter of fibres — Air permeability method

0 INTRODUCTION

When a current of air is passed through a uniformly arranged mass of fibres packed in a chamber with perforated ends, the ratio of air flow to differential pressure is uniquely determined by the total surface area of the fibres, and by various constants. This was predicted from the hydrodynamic equations of Kozeny and others.

For fibres of circular or near-circular cross-section and constant density, such as non-medullated wool, the surface area of a given mass of fibres is proportional to the average fibre diameter. This principle can be utilized to construct apparatus giving an estimate of fibre diameter. Because of its speed and simplicity, the method is particularly suitable for quality control in mill testing laboratoires.

Since the method is indirect, the apparatus must first be calibrated from wools of known fibre diameter. For this purpose, eight reference slivers have been provided (see annex E).

It has been shown that the estimate of fibre diameter actually given by the permeability method is $d(1 + c^2)$, where d is the average fibre diameter (length biassed) measured by the projection microscope, and c is the fractional coefficient of variation. Since c normally lies within comparatively small limits for unblended slivers, it is usual, however, to calibrate the apparatus directly in terms of d.

The method requires that the fibres be reasonably clean and dispersed in a uniform open state, such as card slivers or combed slivers. It is thus unsuitable for raw wool unless first scoured and carded. Some types of wool may need special calibrations as described in annex D.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the mean diameter of wool fibres, using an apparatus which passes a current of air through a bundle of fibres.

The method is applicable to clean, unmedullated wool fibres dispersed in a uniform, open state. It is particularly suitable for combed slivers. The method is also applicable to oil-combed slivers without cleaning, if the oil content is constant and the apparatus suitably calibrated.

The method is less accurate for lambswool and for wool which is appreciably medullated (see annex D).

2 REFERENCE

ISO 139, *Textiles — Standard atmospheres for conditioning and testing.*

3 DEFINITIONS

For the purposes of this International Standard, the following definitions shall apply :

3.1 laboratory sample : The conditioned sample of fibres, representative of the bulk, from which the test specimens are weighed out. In many cases the laboratory sample will consist of one or more short lengths of sliver.

3.2 test specimen : The weighed amount of fibre which is packed into the constant volume chamber.

4 PRINCIPLE

A specified mass of fibres to be tested is compressed to a constant volume in a cylindrical chamber with perforated ends to which a flowmeter and a manometer are connected. The fibres are packed in such a way that they lie predominantly at right angles to the long axis of the chamber. A regulated current of air is then passed through the compressed fibres and the average fibre diameter read off from a scale on the manometer or the flowmeter.

5 APPARATUS

5.1 Forms of apparatus

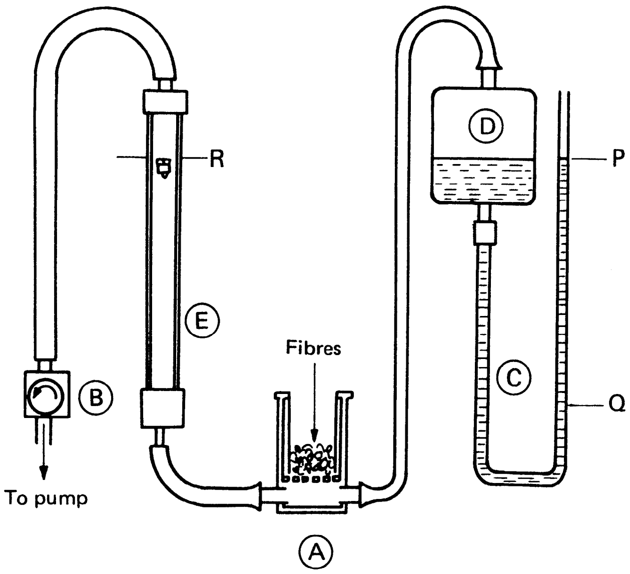
Two alternative forms of apparatus are described : "constant flow" and "constant pressure". Both forms of apparatus have the same arrangement of parts, as illustrated in figure 1.

The constant flow apparatus utilizes a specimen mass of 1,5 g; the flowmeter is adjusted to a fixed value and the fibre diameter is read off from the manometer scale. This scale is not linear since the successive intervals, corresponding to 1 μm, decrease with the diameter.

The constant pressure apparatus utilizes a specimen mass of 2,5 g; the monaometer is adjusted to a fixed pressure and the fibre diameter is read off from the flowmeter. The constant pressure apparatus gives a nearly linear scale in micrometres. Since less accuracy in weighing the specimen is required, this method has some advantages for mill use.

5.2 Detailed parts

The apparatus consists of the following parts arranged as shown in figure 1.



Ⓐ	Constant volume chamber
Ⓑ	Air valve
Ⓒ	Manometer
Ⓓ	Reservoir
Ⓔ	Flowmeter
P, Q, R	Reference marks

FIGURE 1 – **General arrangement of apparatus**

5.2.1 Air valve (B), giving sufficiently fine control of the air supply, such that the lever of the flowmeter or manometer may be quickly adjusted to the working value.

5.2.2 Suction pump, of a type providing a smooth output of at least 30 l/min at 200 mmH_2O* with minimal fluctuation of the float of the flowmeter. A filter to trap any loose fibres may be inserted between the pump and the air valve (B).

5.2.3 Constant volume chamber (A), of brass, hardened steel, or any other suitable metal, comprising the three following parts : the base into which the fibres are packed, the plunger which compresses the fibres, and the screw cap which clamps the plunger to the base. The finish shall be smooth so that the plunger slides easily into the base without trapping fibres.

Suggested dimensions of the constituent elements of the chamber are given in figure 2.

5.2.4 Manometer (C), made of glass tubing of internal diameter at least 5 mm to reduce surface tension effects. In both cases a small amount of dye may be added to the manometer fluid, and where this consists of distilled water, a small trace of chromic acid should be added to give a clear meniscus. A millimetre scale is fixed behind the open limb as described in A.3.1 of annex A.

5.2.5 Reservoir (D) of the fluid manometer (5.2.4), having the characteristics specified in the following table, and mounted at a sufficient height to give a clear working distance PQ of 350 mm in the open limb of the manometer.

TABLE – **Manometer and flowmeter characteristics**

Characteristic	Constant flow	Constant pressure
Minimum diameter of reservoir	150 mm	60 mm
Type of manometer fluid	n-Propyl alcohol	Distilled water
Working range of flowmeter	10 to 20 l/min	5 to 25 l/min

5.2.6 Flowmeter (E), having the characteristics indicated in the table in 5.2.5.

5.2.7 Rubber tube, connecting the manometer reservoir (D) to the chamber (A), consisting of pressure tubing of small internal diameter to avoid constriction at the bends.

5.2.8 Rubber or plastic tube from the chamber (A) to the flowmeter (E), of internal diameter not less than 6 mm. The tube shall be as short as possible and shall not be twisted or kinked between calibration of the apparatus and its subsequent use.

5.3 Balance, capable of weighing the specimen to an accuracy of ± 2 mg for the constant flow method and of ± 4 mg for the constant pressure method.

6 CONDITIONING AND TESTING ATMOSPHERE

6.1 Whenever possible, the laboratory sample shall be conditioned to equilibrium and tested in one of the standard atmospheres for testing specified in ISO 139.

6.2 If tests are not carried out in the standard atmosphere for testing, the laboratory sample shall be conditioned to equilibrium near the apparatus and the relative humidity of the atmosphere at the time of test noted. The final results shall be corrected by the factors given in annex C.

NOTE – A source of error may occur if the moisture of the specimen changes during test. This may happen if the laboratory sample is allowed insufficient time to attain moisture equilibrium with the testing atmosphere. The minimum time required to ensure conditioning to equilibrium of a length of sliver in an opened-out state in a well-ventilated room is about 60 min.

---

* 1 mmH_2O = 9,806 65 Pa = 9,806 65 N/m^2

Dimensions in millimetres

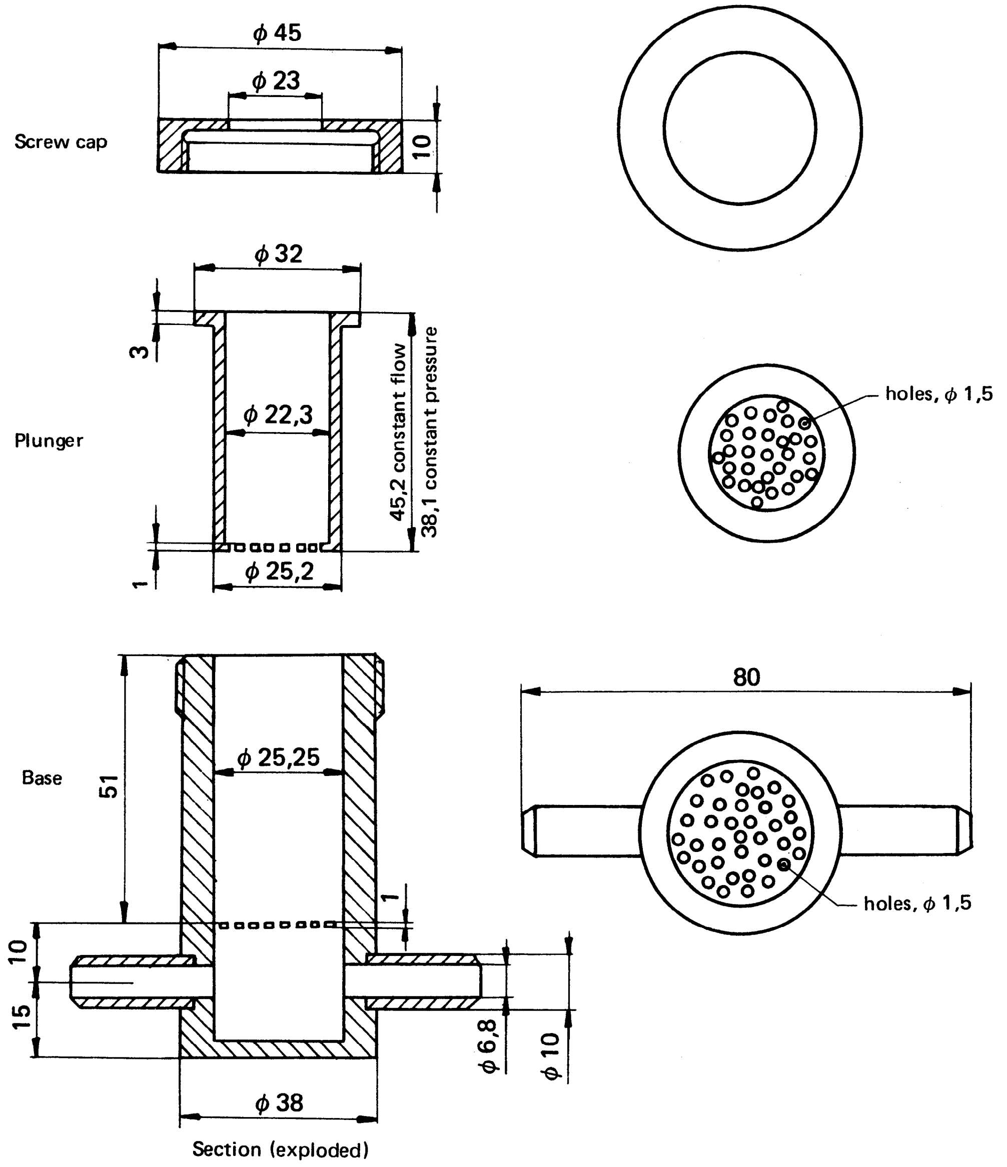


FIGURE 2 – **Suggested dimensions of constant volume chamber (A)**
Important dimensions are 22,3 – 25,2 – 25,25 – 42,5 and 38,1 mm

7 PREPARATION OF TEST SPECIMENS

7.1 Cleaning

In general the laboratory sample shall have a mass of about 8 g and shall first be degreased by rinsing well in two baths each of about 20 ml of petroleum ether before conditioning.

If the laboratory sample is known to be dry-combed with fatty matter content below 1 % (*m/m*), the test specimens may be taken from it without cleaning.

If the laboratory sample is known to be oily with fatty matter content between 3 and 4 % (*m/m*), the test specimens may be taken from it without cleaning provided the apparatus has been calibrated from oil-combed slivers.

7.2 Number of specimens

Unless otherwise specified, test a minimum of two specimens for fibre diameter below 30 μm and a minimum of three specimens for fibre diameter above 30 μm.

7.3 Selection of specimens

The specimens shall be taken from different places in the laboratory sample. In the case of balls of sliver, the laboratory sample shall be made up of pieces of sliver from both inside and outside the ball.

7.4 Specimen mass

For the constant flow method, the specimen mass shall be 1,5 ± 0,002 g. For the constant pressure method, the specimen mass shall be 2,5 ± 0,004 g.

7.5 Preparation

For slivers with cut ends, the specimen shall be prepared by cutting off with scissors a length to give as nearly as possible the specimen mass and then making up to the exact mass by adding shorter cut lengths or portions.

For slivers with pulled ends, about five hand draws shall be removed and discarded and the specimens weighed out by taking several successive hand draws.

These two methods of sampling give the same results if carried out properly.

8 PROCEDURE

8.1 Ensure that the meniscus of the manometer is at the zero mark and if required carry out an orifice plate check as detailed in A.3.3 of annex A.

8.2 Pull out the weighed test specimen into a long thin sliver and feed it evenly into the constant volume chamber (A), packing the fibres down with a smooth rod from time to time. Insert the plunger and screw down the cap to the furthermost extent so that the lip of the plunger is in contact with the base.

8.3 Depending on the method to be used, adjust the air valve as follows :

a) for the constant flow method, adjust the air valve until the top of the float of the flowmeter coincides with the reference mark R and note the fluid level of the manometer to the nearest 1 mm or 0,1 μm (see A.3.1 of annex A);

b) for the constant pressure method, adjust the air valve until the fluid level of the manometer coincides with the 180 mm reference mark P and note the position of the float of the flowmeter to the nearest 1 mm or 0,1 μm (see A.3.2 of annex A).

8.4 Remove the specimen from the constant volume chamber, tease out the fibres by hand, repack in the constant volume chamber without loss of fibre, insert the plunger and screw down the cap.

8.5 Repeat the operation specified in 8.4 so that a total of three readings on each test specimen is obtained.

9 EXPRESSION OF RESULTS

Calculate the average of the three readings for each specimen and express the result to the nearest 0,1 μm.

10 TEST REPORT

The test report shall include the following particulars :

a) the reference to this International Standard;

b) the method used (constant flow or constant pressure);

c) the results obtained in accordance with clause 9;

d) whether the sample was tested after cleaning in petroleum ether or without cleaning;

e) the relative humidity and temperature of the conditioning and testing atmospheres and whether the result has been corrected for the relative humidity;

f) all operating conditions not specified in this International Standard, as well as any incidents that may have influenced the results.

ANNEX A

CALIBRATION OF APPARATUS

A.1 LEAKAGE TEST

After assembling the apparatus as in figure 1, remove the cap and plunger from the constant volume chamber (A) and insert a rubber stopper. By means of a Hoffmann clip, close the rubber tube between (A) and (E) after introducing a pressure difference causing the level of the meniscus in the manometer to alter by about 150 mm. Note the position of the meniscus periodically for several minutes; if it changes, examine the apparatus for leaks.

A.2 SAMPLES OF SLIVERS

Obtain sufficient quantities of the reference slivers (see annex E) for calibration. In requesting these, state

a) the test specimen mass for the apparatus to be used (1,5 or 2,5 g);

b) whether oil-combed or dry-combed samples are required.

Sufficient numbers of each type of sliver are normally supplied for four specimens.

A.3 GRADUATING THE SCALE

A.3.1 Constant flow apparatus

Make a horizontal mark R (see figure 1) near the top of the flowmeter scale, avoiding any position giving marked fluctuation of the float. Fix a scale graduated in millimetres behind the manometer and adjust the zero mark to coincide with the meniscus of the liquid. Then condition and weigh out, according to the procedure specified in clauses 6 and 7, 1,5 g specimens of each sample of reference sliver and test according to the procedure specified in clause 8, noting the distance in millimetres below the zero to which the meniscus falls. Do not clean the sliver before test. Test three specimens from each of the eight reference slivers in this way and calculate the average of the nine readings for each reference sliver.

Plot the average depression h, in millimetres, of the manometer meniscus against the known value of fibre diameter d, in micrometers, and, after inspection to ensure that the points lie about a smooth curve, fit a relation by least squares as given below. From this relation a conversion table may be prepared, in micrometres, or a scale may be graduated in micrometres and fixed behind the manometer.

Adjustment of results by the least squares method

The relation between d and h is of the form hd^b = constant and it is thus necessary to take logarithms to obtain a linear relation.

Let $X = \log d$ and $Y = \log h$.

For each of the n lots of sliver used for standardization, two values (X_1 and X_2, X_2 and Y_2, etc.) are obtained.

First calculate the following quantities :

$$\Sigma X = X_1 + X_2 + \dots + X_n$$

$$\Sigma Y = Y_1 + Y_2 + \dots + Y_n$$

$$\Sigma Y^2 = Y_1^2 + Y_2^2 + \dots + Y_n^2$$

$$\Sigma XY = X_1 Y_1 + X_2 Y_2 + \dots + X_n Y_n$$

$$\Sigma y^2 = \Sigma Y^2 - \frac{(\Sigma Y)^2}{n}$$

$$\Sigma xy = \Sigma XY - \frac{\Sigma X \, \Sigma Y}{n}$$

$$b = \frac{\Sigma xy}{\Sigma y^2}$$

The regression equation of X and Y which applies to the apparatus is then

$$X = \frac{\Sigma X}{n} + b\left(Y - \frac{\Sigma Y}{n}\right) \qquad \dots (1)$$

Finally, construct a table relating h to d by taking values of h at 5 mm intervals, finding $\log h$, substituting in equation (1) to obtain X and so tabulating $d = \text{antilog}\, X$ for each value of h.

A.3.2 Constant pressure apparatus

Make a horizontal mark at a distance corresponding to 180 mm water pressure from the zero mark Q of the manometer. Fix a scale graduated in millimetres behind the flowmeter (E) so that the zero of this scale coincides with a file mark (zero) made near the bottom of the flowmeter. Condition and weigh out 2,5 g specimens of each sample of reference sliver according to the procedure specified in clauses 6 and 7, and test according to the procedure specified in clause 8, noting the distance y, in millimetres, of the float of the flowmeter from zero. Do not clean the slivers before test. Test three specimens from each of eight reference slivers in this way and calculate the average of the nine readings for each reference sliver.

Plot the average reading, in millimetres, y_1, y_2, etc., against the known values of fibre diameter d_1, d_2, etc. The result will be a nearly linear relation; fit a second degree regression line of y on d. This is done by finding the coefficients a, b, c, in the equation

$$y = a + bd + cd^2 \qquad \ldots (2)$$

by solving the equations

$$\Sigma y = 8a + b\, \Sigma d + c\, \Sigma d^2$$

$$\Sigma dy = a\, \Sigma d + b\, \Sigma d^2 + c\, \Sigma d^3$$

$$\Sigma d^2 y = a\, \Sigma d^2 + b\, \Sigma d^3 + c\, \Sigma d^4$$

The equation (2) is then used to graduate a scale, in micrometres, which may be fixed behind the flowmeter.

A.3.3 Orifice plate checks

To make regular daily checks that the apparatus is in good order, the use of the two orifice plates is recommended. These consist of aluminium disks of the same diameter as the inside of the constant volume chamber, each with a central hole. The disks have a rim which in use rests on the annular top of the constant volume chamber. The diameter of the central hole in one disk is chosen to give a reading of about one-third of the available scale on the manometer (constant flow method) or flowmeter (constant pressure method) when clamped and used in the apparatus under working conditions, with no fibres in the chamber. The diameter of the central hole in the second disk is chosen to give a reading of about two-thirds of the available scale under the conditions described above.

At least once a day, clamp the orifice plates in the apparatus so that air enters through the central hole only and note the readings. Variations in the readings given by the scale shall not exceed 2 mm and 4 mm respectively for the two orifice plates. This provides a useful and quick check on the functioning of the apparatus, particularly as regards the presence of air bubbles in the manometer system.

ANNEX B

REPRODUCIBILITY OF RESULTS

It is desirable that the persons to whom a test result is communicated should have some idea of the appropriate confidence limits of each average reading reported. Confidence limits will depend amongst other things on the number of tests, the variability of the material, experimental error, differences between apparatus, and the probability level assumed. There are two important cases about which information is available at present. This information, which is summarized below, should be regarded as illustrative and as applying only to the particular material tested. The original papers should be consulted for further details.

"Within sample" confidence limits

Suppose about 1 m of sliver is received for test and n test specimens are weighed out and tested, three readings being taken on each in accordance with standard procedure. A total of $3n$ readings would be obtained, and since the variance due to repacking is normally about the same as that between different weighings, the 95 % confidence limits of the average readings are given by

$$\pm \frac{1{,}96\,\sigma}{\sqrt{3n}}$$

where σ is the standard deviation of the $3n$ readings. From the work of various authors, it appears that the value of σ is about 0,2 μm for wool of 20 μm, rising to about 0,4 μm for wool of 30 μm.

"Between apparatus" confidence limits

The following confidence limits have been established by a determination carried out by the usual method on two test specimens in any one of 16 laboratories participating in tests with certified apparatus using parts of four identical reference slivers.

Average μm	**95 % confidence limits** μm
20	± 0,18
25	± 0,29
30	± 0,42
35	± 0,59

Variability within lots during processing

Although the variability within lots is not related to the reproducibility of the method of test, it is sometimes necessary to take variability within lots into account when comparing results obtained in different laboratories, since the laboratory samples may have been taken at different times from different portions of a non-homogeneous lot. Some tests relating to variations in mean fibre diameter of combed slivers during processing have shown that significant differences may occur.

ANNEX C

CORRECTION FOR RELATIVE HUMIDITY

The temperate standard conditions for testing as specified in ISO 139 are 65 ± 2 % relative humidity and a temperature of 20 ± 2 °C. If tests are carried out in a non-standard atmosphere of known relative humidity, the results in micrometres may be corrected by the following factors, applicable to fibre diameters between 19 and 37 μm.

Relative humidity %	**Multiplier to convert to 65 % relative humidity**
40	1,022
45	1,019
50	1,015
55	1,010
60	1,005
65	1,000
70	0,995
75	0,988
80	0,980
85	0,969

ANNEX D

SPECIAL TYPES OF WOOL

Noils

It has been shown that in general the air-flow method is applicable to noils. Particular care should be taken, however, when testing noils to remove all vegetable matter and to degrease the laboratory sample with petroleum ether before weighing out the specimens. Representative laboratory samples should be made up by taking bunches of fibres from several places in the bulk.

Lambswool

Tests have shown that the estimate of diameter obtained by the air-flow methods were systematically lower than those given by the projection microscope, the apparatus having been calibrated from slivers of ordinary wool.

The maximum difference obtained in estimated fibre diameter by the two methods was 6,7 %.

Medullated wool

The theory of the permeability (air-flow) method assumes that the fibres have a constant overall density, so that a fixed mass of fibres of the same average diameter will always give the same amount of fibre surface. Highly medullated fibres may have an appreciably lower fibre density than the accepted value of 1,30 to 1,31 g/cm^3 for solid wool fibres. An air-flow test carried out on such medullated fibres will give a reading lower than the fibre diameter as measured on a projection microscope. This error may be of significance for fibre coarser than about 35 μm. The effect is illustrated by the following data taken from a specific study :

Density (g/cm^3)	1,31	1,29	1,27	1,25
Mean apparent fibre diameter obtained by the permeability method (μm)	35,0	34,2	33,2	32,3

Dyed fibres

Differences in mean apparent fibre diameter between undyed slivers and dyed slivers have been reported by various workers when tested by the air-flow method, the dyed fibres giving higher values. The effect is likely to be of significance only for heavier shades, for example chrome black where the difference may be up to about 0,8 μm.

ANNEX E

REFERENCE SLIVERS FOR CALIBRATION

For calibration of the air-flow apparatus as described in annex A, laboratory samples of eight reference slivers are available. The fibre diameter of each sliver has been measured by the projection microscope in several laboratories and is known accurately.

The slivers are available in two forms :

a) dry-combed, fatty matter content less than 1 % (*m*/*m*);

b) oil-combed, fatty matter content between 3 and 4 % (*m*/*m*).

A set of eight reference slivers can be obtained by appropriate laboratories on application to :

International Wool Textile Organization
Box 13
Rue du Luxembourg 19-21
1041 BRUSSELS
Belgium

All such applications should state :

1) whether oil-combed or dry-combed slivers are required, and

b) whether specimen masses of 1,5 to 2,5 g are required.

This page intentionally left blank

This page intentionally left blank